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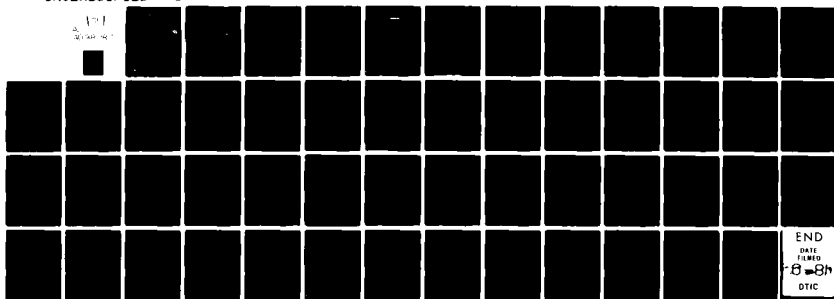
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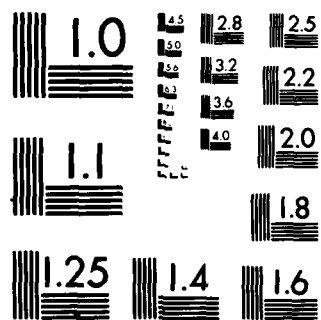
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SECOND SYMPOSIUM ON APPLIED SURFACE ANALYSIS

by

JOHN T. GRANT

AD A098787

Final Technical Report
1 April 1980 - 1 April 1981

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RESEARCH INSTITUTE
300 College Park
Dayton, Ohio 45469

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by

JOHN T. GRANT

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Surfaces	Surface Chemistry									
Surface Analysis	Surface Physics									
	Surface Properties									
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) <p>This report describes the Second Symposium on Applied Surface Analysis held at the University of Dayton, Dayton, Ohio on 11-13 June 1980. Twenty nine papers were presented at the Symposium, six of them being from invited speakers. Approximately 100 scientists active in the field of surface analysis participated in the Symposium.</p>										

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FOREWORD

This report describes the Second Symposium on Applied Surface Analysis held at the University of Dayton, 300 College Park Avenue, Dayton, Ohio 45469, on 11-13 June 1980, under [REDACTED]. *AFDSR-80-0159*

The Symposium was conducted by the University of Dayton with Dr. John T. Grant of that organization and Dr. T. W. Haas of the Materials Laboratory, Air Force Wright Aeronautical Laboratories as Co-Chairmen. This report was submitted in April 1981.

TABLE OF CONTENTS

SECTION		PAGE
I	INTRODUCTION	1
II	SYMPOSIUM GOALS	2
III	PARTICIPANTS	4
IV	PROGRAM	11
V	SYMPOSIUM CONCLUSIONS	47

SECTION I INTRODUCTION

The Second Symposium on Applied Surface Analysis was held at the University of Dayton, 11-13 June 1980. This Symposium was held to meet a need, namely to show the transition between basic surface science research and applications of this research to areas of Air Force interest. Areas receiving special attention at this Symposium were adhesion, catalysis, thin films, and electronic materials. Other topics discussed included adsorption, wear and the effects of electron and ion beams on surface composition. Approximately 100 scientists active in the field of surface analysis participated in the Symposium. Six scientists presented invited papers at the Symposium. There were 23 contributed presentations. The proceedings of the Symposium are being published in a special issue of the journal "Applications of Surface Science" by North-Holland Publishing Company. A tour of the surface analysis facilities at the Materials Laboratory, Wright-Patterson Air Force Base, was also conducted during the Symposium.

SECTION II SYMPOSIUM GOALS

The Second Symposium on Applied Surface Analysis was held at the University of Dayton, 11-13 June 1980. This Symposium was organized to meet a need, namely, to show the continuing transition between basic research and applications of this research to areas of Air Force interest. Workers engaged in basic research and those engaged in applications of surface science research were brought together at the Symposium in an environment suitable for promoting the maximum possible interaction between such workers.

The Symposium was planned and organized by the University of Dayton, with Dr. John T. Grant of the University of Dayton and Dr. T. W. Haas of the Materials Laboratory, Air Force Wright Aeronautical Laboratories as Co-Chairmen. Six invited speakers were selected to cover specific areas of interest. These speakers and their topics were:

1. J. R. Arthur
Perkin-Elmer, Physical Electronics Division
MOLECULAR BEAM EPITAXIAL GROWTH OF SURFACES, INTERFACES,
FILMS AND DEVICES
2. D. T. Clark
University of Durham
ESCA STUDIES OF POLYMER SURFACES
3. T. E. Fischer
Exxon Research and Engineering Company
CONTRIBUTIONS OF SURFACE SCIENCE IN THE CATALYSIS OF ETHYLENE
OXIDATION AND METHANE SYNTHESIS
4. L. L. Kazmerski
Solar Energy Research Institute
APPLICATIONS OF AES, SIMS AND XPS TO SOLAR ENERGY RESEARCH
5. C. G. Pantano
Pennsylvania State University
ELECTRON BEAM DAMAGE IN AUGER ELECTRON SPECTROSCOPY
6. R. W. Springer
Los Alamos Scientific Laboratory
CHARACTERIZATION, GROWTH AND MECHANICAL PROPERTIES OF
LAMINATE FILMS

Contributed papers on applied surface analysis were also solicited from the research community, resulting in 23 such papers being presented at the Symposium.

It was decided to publish as many of the papers as possible in an appropriate journal, thereby providing a permanent record of the work presented at the Symposium. The journal "Applications of Surface Science" was selected, and the journal editor was pleased to be able to publish such a proceedings. Fourteen manuscripts were submitted for peer review and eventual publication in the proceedings. It is hoped that the proceedings will be published and distributed to all attendees in the spring of 1981.

The Symposium was advertised through appropriate scientific societies, equipment manufacturers, scientific journals, and at other meetings. It was decided to hold the Symposium on the University of Dayton campus and to encourage attendees to stay in University apartments in order to provide an informal atmosphere and to optimize the interaction between attendees.

Two events were organized by the University and held in conjunction with the Symposium, namely a two-day short course and a vendors' exhibit. The short course topic was "Techniques for Surface Analysis", and was held immediately before the Symposium to provide an opportunity for attendees to learn the fundamentals of several surface analysis techniques, so that they could gain as much as possible from the Symposium presentations and in their own research. The vendors' exhibit provided an opportunity for attendees to learn about the commercial surface instrumentation that is currently available and what is planned for the the future.

SECTION III
PARTICIPANTS

Approximately 100 scientists active in the field of surface analysis attended the Symposium.

The list of participants follows.

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SECTION IV PROGRAM

The technical program was divided into five sessions. There were four main technical sessions, each starting with a presentation by an invited speaker. The fifth session began with presentations by manufacturers' representatives, describing developments in surface analysis instrumentation, and concluded with a tour to the surface analysis laboratories at the Materials Laboratory, Wright-Patterson Air Force Base.

The program organization and the abstracts of the papers presented at the Symposium follow.

2nd SYMPOSIUM ON APPLIED SURFACE ANALYSIS

11 - 13 JUNE 1980
University of Dayton

GENERAL INFORMATION

The 2nd Symposium on Applied Surface Analysis is being supported by the Air Force Office of Scientific Research. The Symposium will provide an opportunity for workers in all areas of surface characterization to meet and discuss applications of surface analysis. It is the aim of the chairmen to attract participants from universities, and from Government, nonprofit and industrial laboratories to promote the maximum interaction between workers in different areas.

LOCATION AND DATES

The University of Dayton
300 College Park
Dayton, Ohio 45469
11-13 June 1980

Limousine service is available to downtown Dayton from Dayton International Airport every hour (20 minutes to the hour), for approximately \$4.50. Taxi service from downtown to the campus is approximately \$4. City bus service (#5 and #12, southbound) leaves downtown Third and Main Streets approximately every 15 minutes. The bus stops within two or three blocks of the apartment at 361 Stewart Street and is \$.50. Apartment keys can be secured from the Apartment Caretaker/Manager upon arrival at the apartment. Checkout times on Friday, 13 June are 8:00-9:00 am, 12:00-1:00 pm, and 3:00-4:00 pm.

HEADQUARTERS

The Symposium headquarters will be adjacent to the O'Leary Auditorium located in Miriam Hall on the University of Dayton Campus. Emergency telephone messages can be taken at Area Code 513-229-3118.

REGISTRATION

All symposium attendees should preregister by mail. Those preregistered may pick up their final programs, etc., on Tuesday evening between 7:00-10:00 pm in Apartment 2A, 361 Stewart Street, if they desire. Symposium registration will also be held during the Symposium starting at 8:00 am on Wednesday, 11 June.

SESSIONS

All sessions will be held in O'Leary Auditorium located in Miriam Hall on the University Campus. The Symposium will close at approximately 12:00 noon on Friday, 13 June.

PROCEEDINGS

The proceedings will be published in a special issue of "Applications of Surface Science". Manuscripts should be given to the Symposium Secretary at the Registration Desk.

VENDORS EXHIBITS AND RECEPTION

A vendor's exhibit will be held from 12 noon Wednesday, 11 June through 3:30 pm Thursday, 12 June. Representatives from the major surface instrumentation and ultrahigh vacuum equipment manufacturers will be available to discuss your instrumentation needs. Time will be available on Thursday from 1:00-3:00 pm for vendors to make a formal presentation on their most recent products. An informal reception will be held at 4:30 pm on Thursday, 12 June in the 8th floor lounge, Miriam Hall.

LABORATORY TOURS

Tours will be arranged from 1:00-4:00 pm on Thursday afternoon, 12 June to the surface science laboratories at the University of Dayton and at the Materials Laboratory at Wright-Patterson Air Force Base for those interested. Non-U.S. citizens must arrange clearance in advance through their Embassy for touring the Air Force Base. Bus transportation will be arranged.

ATTIRE

Dayton is known as one of Ohio's most progressive cities. There are many interesting places to visit while in the area. In June, the mean daytime temperature is 78°F to 81°F; the mean evening temperature is 55°F to 62°F. Mid and lightweight dress is recommended.

SYMPOSIUM ADMINISTRATION

SYMPOSIUM CHAIRMEN

John T. Grant
University of Dayton Research Institute
Dayton, Ohio

T. W. Haas
Air Force Wright Aeronautical Laboratory (AFWAL/MLBM)
Wright-Patterson Air Force Base, Ohio

VENDOR COORDINATOR

James R. Hoenigman
University of Dayton Research Institute
Dayton, Ohio

SYMPOSIUM SECRETARY

Patsy R. Collins
University of Dayton Research Institute
Dayton, Ohio

AGENDA

WEDNESDAY MORNING, 11 JUNE 1980

SESSION A

Chairman: Dr. L. L. Kazmerski
Solar Energy Research Institute

- 8:00 REGISTRATION
- 9:00 INTRODUCTORY REMARKS
- A-1 9:20 T. E. Fischer (Invited)
Exxon Research and Engineering Company
CONTRIBUTIONS OF SURFACE SCIENCE IN THE CATALYSIS
OF ETHYLENE OXIDATION AND METHANE SYNTHESIS
- A-2 10:10 G. A. Sargent and J. Chao
University of Kentucky, and
G. Freeman
Kentucky Center for Energy Research Laboratory
THE ADSORPTION OF CO ON AND S POISONING OF A Ni(111)
SINGLE CRYSTAL WITH SMALL ANGLE BOUNDARIES
- 10:30 COFFEE BREAK
- A-3 10:50 M. L. Yu and W. Reuter
IBM T. J. Watson Research Center
TWO GENERAL RULES FOR THE OXYGEN ENHANCEMENT OF SECONDARY
ION YIELDS FROM BINARY ALLOYS
- A-4 11:10 R. P. Burnger, T. R. Lundquist, and P. R. Swann
Gatan, Inc., and
I.S.T. Tsong
Pennsylvania State University
HYDROGEN DEPTH - PROFILING USING SIMS AND SIPS
- A-5 11:30 B. F. Phillips and R. L. Gerlach
Perkin Elmer Corporation, Physical Electronics Division
APPLICATIONS OF HIGH SENSITIVITY QUADRUPOLE SIMS ANALYSIS
- A-6 11:50 T. N. Wittberg, J. D. Wolf, and R. S. Harmer
University of Dayton Research Institute
SOME COMPARATIVE SURFACE STUDIES OF TWO TYPES OF NICKEL
MATRIX CATHODE
- A-7 12:10 N. C. Fernelius, D. V. Dempsey, and D. B. O'Quinn
University of Dayton Research Institute
PHOTOACOUSTIC AND LASER RATE CALORIMETRY STUDIES OF THE
BULK AND SURFACE OPTICAL ABSORPTION OF WINDOW MATERIALS
- 12:30 LUNCH

WEDNESDAY AFTERNOON, 11 JUNE 1980

SESSION B

Chairman: Dr. C. G. Pantano
Pennsylvania State University

- B-1 1:30 J. R. Arthur (Invited)
Perkin-Elmer, Physical Electronics Division
MOLECULAR BEAM EPITAXIAL GROWTH OF SURFACES, INTERFACES,
FILMS AND DEVICES
- B-2 2:20 D. E. Savage and M. G. Lagally
University of Wisconsin, and
M. E. Schrader
David Taylor Naval Ship Research and Development Center
STUDIES OF CARBON BRUSH WEAR USING SURFACE ANALYTICAL
TECHNIQUES
- B-3 2:40 D. L. Jones
Tektronix Laboratories
A SURFACE ANALYTICAL STUDY OF CORROSION ON COPPER-TIN-
ZINC PLATED ALUMINUM
- 3:00 COFFEE BREAK
- B-4 3:20 L. L. Kazmerski (Invited)
Solar Energy Research Institute
APPLICATIONS OF AES, SIMS AND XPS TO SOLAR ENERGY RESEARCH
- B-5 4:10 W. L. Baun
Air Force Wright Aeronautical Laboratories
CHARACTERIZATION OF HgCdTe AND OTHER COMPOUND
SEMICONDUCTORS WITH ISS
- B-6 4:30 J. Morar, T. L. Einstein, and R. L. Park
University of Maryland
SHORT RANGE ORDER OF Si/O SURFACES
- B-7 4:50 J. A. Taylor
Perkin-Elmer Corporation, Surface Sciences Division
FURTHER EXAMINATION OF THE Si(KLL) AUGER LINE IN
SILICON NITRIDE THIN FILMS
- 5:10 ADJOURNMENT

THURSDAY MORNING, 12 JUNE 1980

SESSION C

Chairman: Dr. J. R. Arthur
Perkin Elmer, Physical Electronics Division

- C-1 8:30 C. G. Pantano (Invited)
Pennsylvania State University
ELECTRON BEAM DAMAGE IN AUGER ELECTRON SPECTROSCOPY
- C-2 9:20 F. Ohuchi and P. H. Holloway
University of Florida
APPLICATION OF RUTHERFORD BACKSCATTERING TO ANALYSIS OF GLASSES
- C-3 9:40 S. W. Gaarenstroom
General Motors Research Laboratories
PRINCIPAL COMPONENT ANALYSIS OF AUGER LINE SHAPES AT SOLID-SOLID INTERFACES
- C-4 10:00 G. E. Hammer
The Goodyear Tire and Rubber Company
AES DEPTH PROFILING OF TRACE ELEMENTS IN A MULTIPLE TRANSITION METAL SYSTEM USING SPECTRUM SUBTRACTION TECHNIQUES
- 10:20 COFFEE BREAK
- C-5 10:40 R. W. Springer (Invited)
Los Alamos Scientific Laboratory
CHARACTERIZATION, GROWTH AND MECHANICAL PROPERTIES OF LAMINATE FILMS
- C-6 11:30 D. R. Baer
Pacific Northwest Laboratory
PROTECTIVE AND NON-PROTECTIVE OXIDE FORMATION ON 304 STAINLESS STEEL
- C-7 11:50 R. J. Koch, P. S. Habig, and F. M. Kilbane
ARMCO, Inc.
SURFACE SEGREGATION IN HOT-DIP METALLIC COATINGS ON STEEL SUBSTRATES
- 12:10 LUNCH

THURSDAY AFTERNOON, 12 JUNE 1980

SESSION D

- 1:10 VENDORS PRESENTATIONS
Program available at symposium
- TOURS - MATERIALS LABORATORY, WRIGHT-PATTERSON AIR FORCE BASE
AND UNIVERSITY OF DAYTON SURFACE SCIENCE LABORATORY
- 4:30 INFORMAL RECEPTION - 8th Floor Lounge, Miriam Hall

FRIDAY MORNING, 13 JUNE 1980

SESSION E

Chairman: Dr. R. W. Springer
Los Alamos Scientific Laboratory

- E-1 8:30 D. T. Clark (Invited)
University of Durham
ESCA STUDIES OF POLYMER SURFACES
- E-2 9:20 H. G. Tompkins and S. P. Sharma
Bell Telephone Laboratories
ON THE THICKNESS AND SPATIAL DISTRIBUTION OF A FLUOROPOLYMER
FILM DEPOSITED BY SOLUTION DIPPING
- E-3 9:40 J. P. Delrue, M. Chtaib, and R. Caudano
Facultés Universitaires
THE USE OF ELECTRONIC DEVICES IN AN ATTEMPT TO STIMULATE
CATALYTIC REACTIONS
- E-4 10:00 B. A. Quimby and S. R. Shatynski
Rensselaer Polytechnic Institute
THE KINETICS OF THE INITIAL OXIDATION OF NICKEL USING
RESISTIVITY MEASUREMENTS
- 10:20 COFFEE BREAK
- E-5 10:40 A. R. Madura and E. J. Giacomelli
Lord Corporation
INVESTIGATION OF BONDING AND ADHESION PHENOMENA BY COMBINED
ISS/SIMS ANALYSIS
- E-6 11:00 F. J. Boerio and J. W. Williams
University of Cincinnati
STRUCTURE AND PROPERTIES OF ORGANOSILANE PRIMERS FOR
ADHESIVE BONDING
- E-7 11:20 N. T. McDevitt
Air Force Wright Aeronautical Laboratories, and
J. S. Solomon
University of Dayton Research Institute
THIN ANODIC OXIDE FILMS ON ALUMINUM ALLOYS AND THEIR
POTENTIAL CORROSION CHARACTERISTICS
- E-8 11:40 A. A. Roche
Universal Energy Systems, Inc.
W. L. Baun
Air Force Wright Aeronautical Laboratories, and
J. S. Solomon
University of Dayton Research Institute
PHOSPHATE-FLUORIDE ETCHING EFFECTS ON TITANIUM ALLOY SURFACES
- 12:00 SYMPOSIUM CLOSING

CONTRIBUTIONS OF SURFACE SCIENCE IN THE CATALYSIS
OF ETHYLENE OXIDATION AND METHANE SYNTHESIS

T. E. Fischer
Exxon Research and Engineering Co.
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Industrial processes usually involve high gas pressures and finely dispersed catalysts on porous supports. The analytical methods of surface science are based on the interaction of electrons, ions and light with flat surfaces, usually in vacuum. Obviously then, surface science should not be used to simulate industrial processes, but rather to complement the classical methods of catalysis research and extend their capabilities.

In ethylene oxidation catalysis, the challenge is to increase the selectivity of the process by suppressing total combustion of the reagent. The advantage of surface science resides in its ability to isolate the chemistry of atomic adsorbed oxygen which is known to be responsible for ethylene combustion. It is thus possible to observe isolated combustion reactions and to identify the most active ones among the many that had been proposed in the literature and, thereby to guide future research along the most profitable direction (I. Wachs and S. R. Kelemen).

In methane synthesis, surface science allowed to extend the pressure range of reaction kinetics measurements and also to study the reaction in the simpler regime of a single synthesis product. These studies resolved an apparent contradiction in existing measurements and, combined with the consideration of surface concentrations of adsorbates, to build a quantitative model that was verified by experiment (R. S. Polizzotti and J. A. Schwarz).

Wednesday, 9:20 am

A-1

THE ADSORPTION OF CO ON AND S POISONING OF A Ni(111)
SINGLE CRYSTAL WITH SMALL ANGLE BOUNDARIES

By

Gordon A. Sargent*, G. Freeman⁺ and J. Chao*

*Metallurgical Engineering and
Materials Science Dept.
University of Kentucky
Lexington, Kentucky 40506

⁺Institute for Mining and Minerals Research
Kentucky Center for Energy Research Laboratory
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Lexington, Kentucky 40583

ABSTRACT

The adsorption of CO on a Ni(111) single crystal with small angle boundaries was studied using the techniques of Low Energy Electron Diffraction (LEED) and Auger Electron Spectroscopy (AES). The data was compared with that obtained for the adsorption of CO on a perfect Ni(111) single crystal.

The Auger spectra showed that the boundary lines at the crystal surface provided favorable sites for the adsorbed CO to dissociate at temperatures as low as 25°C. The carbon peak shape indicated that the dissociated carbon was primarily in the form of nickel carbide.

Heating the crystal to 850°C caused bulk sulfur impurities to diffuse to the surface. The boundary enhanced dissociation of adsorbed CO was not observed after the diffusion of sulfur to the crystal surface. AES depth profiling of the sulfur concentration at several different positions on the crystal both at the boundaries and between the boundaries gave no evidence that the boundary lines provided an enhanced path for sulfur diffusion from the bulk of the crystal.

Wednesday, 10:10 am

A-2

Two General Rules for the Oxygen Enhancement of Secondary Ion Yields from Binary Alloys.

Ming L. Yu and Wilhad Reuter
IBM T. J. Watson Research Center
Yorktown Heights, NY 10598

It is well known that the presence of oxygen in the sample can enhance positive ion yields by several orders of magnitude in SIMS analysis. However, this oxygen enhancement is a function of sample composition. We have made a systematic SIMS and ESCA study of the oxygen enhanced emission of positive secondary ions from binary alloys. Two rules are established. These rules are generalized from our study of five alloy systems: Cr-Ni, Fe-Ni, Cu-Ni, Pd-Ni, and Ag-Pd. For an alloy A-B, where A forms a stronger oxide bond than B,

- i) Rule I states that the presence of A enhances the emission of B^+ and the presence of B suppresses the emission of A^+ .
- ii) Rule II states that the presence of A sharpens the energy distribution of B^+ while the presence of B broadens the energy distribution of A^+ .

The experiment was performed in an ultrahigh vacuum system with a 15 keV, mass analyzed, differentially pumped ion gun. Both O_2^+ bombardment and Ar^+ bombardment in O_2 ambient were used. Yield enhancements due to alloying can be as large as 10 or more. The widths of the energy distributions can change by as much as 50%. The experimental data fit the rules with better consistency when the oxygen was incorporated in the sample by adsorption rather than by implantation.

In situ ESCA studies of the oxidized alloy surfaces revealed that these observations are linked to the enhanced oxidation of B when more A is present and similarly to the suppressed oxidation of A when more B is present. Rule II is also found to be the consequence of a secondary ion energy dependent ionization probability. The few exceptions (Cu in Cu-Ni alloys) to the rules are related to the failure to incorporate sufficient amount of oxygen on the sample, e.g. because of a low oxygen sticking coefficient. Examples of the effect of these rules on the depth profiling of metallic interfaces will be shown.

Wednesday, 10:50 am

A-3

HYDROGEN DEPTH - PROFILING USING
SIMS AND SIPS

R.P. Burnger, T.R. Lundquist and P.R. Swann
Gatan, Inc.
780 Commonwealth Drive
Warrendale (Pittsburgh), PA 15086

I.S.T. Tsong
Materials Research Laboratory
Pennsylvania State University
University Park, PA 16802

Abstract:

A new SIPS - SIMS scanning ion probe has been developed which can determine depth-profiles of hydrogen in solids. The hydrogen is simultaneously analysed by sputter induced photon spectrometry (SIPS) of excited neutral atoms and by secondary ion mass spectrometry (SIMS) of sputtered ions. In this communication both the SIPS - SIMS instrument and the techniques used for analysis and quantification of hydrogen are described in detail.

Wednesday, 11:10 am

A-4

Applications of High Sensitivity Quadrupole SIMS Analysis

Bradway F. Phillips and Robert L. Gerlach
Perkin Elmer Corporation, Physical Electronics Division
6509 Flying Cloud Drive
Eden Prairie, Minnesota 55344

The ability to perform SIMS analysis on specimens already being analyzed by Auger or ESCA in order to confirm the existence and distribution of elements present at low concentration is of great importance. With the high sensitivity of the SIMS II, this task becomes much easier because the moveable ion extraction lens allows ions to be collected efficiently from small areas on the surface of a specimen. The spherical sector energy analyzer and high transmission quadrupole combined with an off axis particle detector make the SIMS II a truly high efficiency accessory SIMS unit.

As an example, the field of mineral analysis, the occurrence of many elements at low concentration can act as identifiers of ore bodies or indicators of the possibility of other minerals in the surrounding area. Also, the analysis of surface layers a few angstrom thick on freshly fractured minerals can show the concentration of some elements on the fracture planes. The analysis of a feldspar mineral showing these properties will be presented.

In semiconductor materials analysis, not only diffusions and implantations, but interfaces and surface impurities are important. The ability to profile through interfaces using the combined techniques of Auger and SIMS can provide a much more complete picture of the dynamic movement of elements than just one technique alone. Examples of semiconductor surface materials analysis as well as interface profiles will be used to illustrate this point.

Wednesday, 11:30 am

A-5

SOME COMPARATIVE SURFACE STUDIES OF TWO TYPES OF NICKEL MATRIX CATHODE

by

T. N. Wittberg, J. D. Wolf, and R. S. Harmer
University of Dayton Research Institute
Dayton, OH 45469

The surface composition and microstructure have been studied for both rolled nickel matrix (Medicus) cathodes and coined nickel matrix cathodes, the latter having alkaline earth peroxides substituted for the usual alkaline earth carbonate starting materials. While an earlier study has shown the formation of distinct alkaline earth oxide-rich regions on Medicus cathodes during activation, these regions have not been observed for activated coined peroxide cathodes. A correlation between thermionic emission current and surface composition shows that for both types of cathode those exhibiting good emission characteristics had a low ratio of oxygen to total alkaline earth as measured with AES. Some preliminary XPS results have been obtained for a thermally activated Medicus cathode. The XPS results do not provide direct evidence, however, for the existence of a barium monolayer as predicted in some models of thermionic emission. The oxygen XPS data suggests that surface hydroxyl groups are present on the surface of an activated Medicus cathode.

Wednesday, 11:50 am

A-6

Photoacoustic and Laser Rate Calorimetry Studies
of the Bulk and Surface Optical Absorption of Window Materials

Nils C. Fernelius, David V. Dempsey, and David B. O'Quinn
University of Dayton Research Institute, Dayton, Ohio 45469

In the design of laser windows it is desirable to know the ratio of the surface optical absorption to bulk optical absorption of a material. When this ratio is known, then the designer knows where to concentrate his efforts to obtain a lower absorbing window. If the bulk absorption predominates, then the emphasis will be on improving purification of starting materials, crystal growth techniques, and forging methods. If the surface is the major contributor, then the emphasis will be placed on improving polishing and cleaning techniques.

Conventional laser rate calorimetry on a single sample with commonly used window dimensions generally yields the quantity

$$\beta_{\text{eff}} = \beta_B + 2\beta_S/\ell$$

where ℓ is the sample thickness in cm, β_B is the bulk optical absorption coefficient in cm^{-1} and β_S is the surface absorption.

One way to obtain the ratio of surface to bulk absorption, $r = \beta_S/\beta_B$, is to obtain a variety of samples with differences. If they are from equivalent parts of a boule and receive identical surface treatments, conditions seldom achieved in reality, then a plot of $\beta_{\text{eff}}\ell$ versus ℓ will yield β_B and β_S .

A second way is to measure a long bar sample with a thermocouple located near the center of the sample length. If $1/3 \text{ cm} \lesssim r \lesssim 3 \text{ cm}$ and the arrival time of heating from the surfaces takes longer than that from the bulk, then the temperature rise curve will show two slopes. The initial slope will be proportional to β_B and the latter slope to β_{eff} .

A third technique is to plot the photoacoustic (PA) signal from a sample versus chopping frequency. In extreme limits the signal amplitude is proportional to f^{-n} . For predominantly surface absorption $n=1$ and the signal has a phase lag of 90° with respect to the reference signal. When the bulk is dominant $n=3/2$ with a 45° phase lag. Theoretical analyses of the PA signal using the Bennett-Forman theory show that the PA signal begins to have surface-like character for $r=0.005 \text{ cm}$.

Examples of all three techniques will be given for CaF_2 at $1.3 \mu\text{m}$ wavelength. Experimental and theoretical results on a coated ZnSe window at $10.6 \mu\text{m}$ will also be presented.

Wednesday, 12:10 pm

A-7

MOLECULAR BEAM EPITAXIAL GROWTH
OF SURFACES, INTERFACES, FILMS AND DEVICES

By John R. Arthur

Perkin-Elmer
Physical Electronics Division

MOLECULAR BEAM EPITAXY (MBE), a sophisticated modification of vacuum evaporation, has proven to be a valuable preparative technique for surface studies of semi-conductors. One important feature is the extreme flatness of MBE surfaces due to the two-dimensional layer growth process which is operative. Also, the stoichiometry of compound surfaces can be varied to allow studies of the influence of composition on atomic and electronic structure. Ultra-thin films can be prepared for studies of interface composition and bulk diffusion. This paper will review the recent progress in the use of MBE for surface studies, the properties of interfaces and thin films prepared by MBE, and some examples of unique electronic device structures which take advantage of these properties.

Wednesday, 1:30 pm

B-1

STUDIES OF CARBON BRUSH WEAR USING SURFACE ANALYTICAL TECHNIQUES

D. E. Savage and M. G. Lagally*
Department of Metallurgical and Mineral Engineering
and Materials Science Center
University of Wisconsin, Madison, WI 53706

and

M. E. Schrader
David Taylor Naval Ship Research and Development Center
Annapolis, MD 24102

In most electrical generators and motors, sliding contacts, or brushes, are used to transfer current from moving conductors to stationary ones. Most brushes are made of carbon because of its low friction, good conductivity, and relatively high sublimation point.

Carbon brushes commonly fail because of excessive wear, but the reasons for such failure are not completely understood, and may depend on environment and history. We have investigated the surface properties of both carbon brushes and copper commutators that had failed due to excessive wear. Samples were examined using optical microscopy, scanning electron microscopy (SEM), x-ray emission spectroscopy (EDAX), scanning Auger microscopy (SAM), and x-ray photoelectron spectroscopy (XPS). The first two techniques were used to study surface morphology, and the latter three to give the composition of the surfaces.

Optical studies of the worn Cu commutators showed the presence of white glassy particles, ranging from $10\mu\text{m}$ to $30\mu\text{m}$ in size, as well as a milky film. Wear tracks were also clearly visible with widths between $1\mu\text{m}$ and $10\mu\text{m}$. Particles were also observed on the carbon brushes and wear tracks compared in size with those on the commutators. SEM studies confirmed these results, and x-ray analysis (EDAX) showed the particles to be primarily silicon. EDAX was not able to identify the milky film.

A true surface chemical analysis was obtained using SAM and XPS. SAM with $3\mu\text{m}$ lateral resolution showed that the commutator surface was partially covered with a thin film of SiO_2 . The Si and O Auger maps correlated with each other and anticorrelated with the C map. The SiO_2 was concentrated in the wear valleys, i.e., the flat contact regions were predominantly covered with C while the wear valleys included Si, Cu, O, and Cl. In addition SAM confirmed the existence of SiO_2 particles on the Cu commutators and found what appears to be various salts containing Cu, C, Si, Cl, N, O, Ca, Mg, S, and Fe.

Auger analysis of the carbon brushes showed the presence of C, Cu, Si, O, Na, and Cl. SAM studies confirmed the presence of SiO_2 particles. NaCl particles and patches of copper chloride and copper oxide were also found on the brushes.

XPS studies of both C brushes and Cu commutators confirmed the presence of the chemical compounds and elements observed in AES.

It is known that the presence of silicone vapors can cause excessive wear of carbon brushes.¹ High temperatures are generated in the contact area due to current flow. It is presumed that the silicone is oxidized in this area and that the resulting silica in either particle or film form is responsible for the increased wear. We discuss possible mechanisms for the wear process.

¹J. Marsden and R. H. Savage, AIEE Trans. 67, 1084 (1948)

*H. I. Romnes Fellow

Wednesday, 2:20 pm

A Surface Analytical Study of Corrosion on
Copper-Tin-Zinc Plated Aluminum

Douglas L. Jones
Materials Analysis Laboratory
Tektronix Laboratories
Beaverton, Oregon 97077

An electrodeposited ternary copper-tin-zinc alloy is used on many parts in electronic instruments. It provides surface corrosion protection and maintains a bright silvery appearance. Functionally, it has additional attributes valuable for this application; it is non-magnetic, has low and stable electrical contact resistance, is solderable, and has hardness and abrasion resistance approaching that of chromium.

In this study, surface corrosion is investigated on cast aluminum alloy panels whose final surface plating is a copper-tin-zinc alloy. Auger electron spectroscopy (AES) combined with ion sputter profiling was used to determine the elemental constituent depth distributions in corroded and non-corroded areas and scanning Auger microscopy (SAM) gave elemental spacial distributions. Scanning electron microscopy (SEM) was used to examine the base aluminum casting after profiling and produced key information in confirming the cause of the corrosion.

The initial Auger results indicated a surface corrosion problem, but more complete examination revealed a basic casting flaw which deleteriously effected the subsequent plating processes resulting in the surface corrosion pit.

The results of this study again confirm the value of combining surface analytical techniques in solving problems.

Wednesday, 2:40 pm

B-3

APPLICATIONS OF AES, SIMS AND XPS TO SOLAR ENERGY RESEARCH

L.L. Kazmerski
Photovoltaics Research Branch
Solar Energy Research Institute
Golden, Colorado 80401

The future of solar technologies as viable energy alternatives still depends largely upon the improvement of device/component/collector performance, the reduction of most of the inherent systems costs, and the establishment of component and array reliabilities for reasonable lifetimes (e.g., 10 to 20 years). Whether one considers the active conversion schemes - photovoltaics, thermal, bio/chemical, photoconversion, thermoelectric - or the passive ones, the system's performance, reliability and cost depend upon both the composition and quality of the materials used and, especially, the integrity of the numerous interfaces between material layers. Because of these problem areas, the rate of development and implementation of solar energy conversion may depend directly and critically upon the contributions of those research techniques which are able to provide information about the chemical, compositional and, in some cases, electrical characteristics of layers 30Å or less into a material. The presentation highlights the complementary application of three of these techniques - Auger electron spectroscopy (AES), secondary ion mass spectroscopy (SIMS) and x-ray photoelectric spectroscopy (XPS) - to the solution of performance and stability problems in photovoltaic devices. The strengths and limitation of each of these surface analytical methods are discussed. Special applications to grain boundary and device interface investigations for Si, GaAs and InP solar cells are presented. Results of micro-compositional and chemical analyses are correlated directly with EBIC and special scanning AES measurements performed in-situ on identical sample areas. Correlation to solar cell performance are also presented. The necessity of applying several complementary surface microanalysis techniques for the unambiguous solution of devices related problems is stressed throughout.

Wednesday, 3:20 pm

B-4

CHARACTERIZATION OF HgCdTe AND OTHER COMPOUND
SEMICONDUCTORS WITH ISS

W. L. Baun

Materials Laboratory
Air Force Wright Aeronautical Laboratories (AFWAL/MLBM)
Wright-Patterson Air Force Base, Ohio 45433

Results are shown here for low energy ion scattering spectrometry (ISS) experiments on compound semiconductors such as mercury cadmium telluride (HgCdTe) grown by liquid phase epitaxy (LPE) on CdTe substrates. By proper etching of the substrates and control of epitaxial growth conditions, surfaces of the zinc blende sphalerite structure may be obtained in which {111} faces have a top layer of cadmium and mercury (A sites) or of tellurium (B sites). Ion scattering experiments at low energies (500 V or less) on epitaxial HgCdTe show only A or B sites. However, when the bombarding ion beam potential is raised to values conventionally used (1500-2500 V), then energy losses from both A and B atoms are observed. At these higher energies the ions displace the outer atoms or tunnel through to interact with atoms in the second layer. Spectra obtained at these higher energies show interesting fine structure which suggest that energy losses are occurring in multiple collisions and nonbinary scattering. Scattered ion yield curves, which for some elements show oscillatory fine structure, are dramatically different for A and B surfaces. Even though these yield curves are very complicated because of the structure and number of elements, the fine structure may prove useful for further understanding of interaction of neighboring atoms. Yield curves along with conventional low energy ion scattering spectra provide a unique method of analysis of the outermost atomic layer of epitaxial films. Both chemical and structural information is obtained in other compound semiconductors. Examples are shown of scattered ion yield curves and low energy effects for GaAs, GaSb, GaP and other semiconductor surfaces.

Wednesday, 4:10 pm

B-5

SHORT RANGE ORDER OF Si/O SURFACES*

John Morar, T. L. Einstein, and Robert L. Park
University of Maryland
Department of Physics and Astronomy
College Park, Md. 20742

Fine structure extending hundreds of volts above core level edges in appearance potential spectra has previously been analyzed to determine nearest neighbor interatomic spacings in both overlayer and elemental systems. These measurements were made by measuring the secondary electron yield from electron bombarded samples, a technique which is relatively insensitive to oxygen on Si. We have therefore constructed a novel high sensitivity soft X-ray appearance potential spectrometer employing a nude surface barrier detector and field emission array electron source. With this spectrometer we observe fine structure extending several hundred volts above the oxygen K edge. Such extended fine structure is analogous to that obtained by EXAFS which depends on photons for the excitation of electronic core levels. The use of EXAFS for the study of surface layers is hampered by the weak interaction between photons and matter. In the appearance potential extended fine structure technique electrons are used to excite core levels, thereby rendering the technique intrinsically surface sensitive and thus well suited to the study of surface layers. Although electron impact excitation does not involve rigid dipole selection rules, explicit calculations show electrons ejected from the oxygen 1s level to be overwhelmingly s-wave. Calculated phase shifts for silicon and oxygen were supplied by G.L. Laramore. A comparison of our results with recently published silicon dioxide surface EXAFS measurements will be presented.

*Work supported by the U.S. Office of Naval Research under Contract N00014-75-C-0292. Computer time and facilities at U. of Maryland provided by U. of Maryland Computer Science Center.

Wednesday, 4:30 pm

B-6

Further Examination of the Si(KLL) Auger Line
in Silicon Nitride Thin Films

by

J. Ashley Taylor
Perkin-Elmer Corporation, Surface Sciences Division
6509 Flying Cloud Drive, Eden Prairie, MN 55344

The chemical bonding of Si in silicon nitride and silicon oxynitride thin films has received a good deal of attention using a variety of spectroscopic techniques. In the case of XPS studies, binding energy shifts in the Si 2p line are used, and in the case of AES studies, generally kinetic energy shifts or shape changes in the Si(LVV) line are used. Although chemical shifts are well known for the Si(KLL) (larger than those for the Si 2p), less work focusing on high resolution spectra has been published. We have looked at a number of different silicon nitride films and their oxide layers using XPS and AES. For XPS, the Si(KLL) line is generated by using the Bremsstrahlung continuum which is produced with the Mg K α x-ray line. The Auger spectra are collected by pulse counting (as in XPS) in the retarding mode at constant pass energy yielding constant resolution. The energy resolution of the Si(KLL) (1.3 eV FWHM for Si) is comparable to the Si 2p line when run in this mode for both the x-ray or electron beam excitation. The advantage of using x-ray excitation is that both Auger and photoelectron lines can be obtained with less film damage and charging than with the electron beam excitation. The difference between the Auger and photoelectron lines (the Auger parameter) gives useful chemical bonding information independent of charging. Binding energy shifts, kinetic energy shifts and Auger parameters have been tabulated for Si, thermal SiO₂, silicon nitride films of various Si, N and O compositions, and silicon nitride films formed by N₂⁺ ion bombardment. A number of the Si(KLL) lines have been curve resolved and the differences for different film compositions have been noted. The passivation oxide layer on the silicon nitride is compared with SiO₂ layers on Si. Such analysis of the Auger lines shows some sub-stoichiometric Si-N bonding in some films after sputtering with argon, but no elemental Si is seen. Some of the sub-stoichiometric bonding can be converted to Si₃N₄ bonding by additional N₂⁺ sputtering.

Wednesday, 4:50 pm

B-7

ELECTRON BEAM DAMAGE IN
AUGER ELECTRON SPECTROSCOPY

Carlo G. Pantano
Department of Materials Science & Engineering
The Pennsylvania State University
University Park, PA 16802

It is well known that electron beam irradiation during AES analysis can damage, or otherwise perturb, the sample surface. Sample damage can arise from one of a number of fundamental phenomena including (i) charging, (ii) heating, (iii) electron stimulated desorption (ESD), and (iv) molecular decomposition. ESD is a direct effect influencing adsorbed surface species as well as ionic solids. The consequences of charging, heating and molecular decomposition are less direct, however, but can lead to ion migration, evaporation of volatile species, adsorption of residual gases, oxidation and electronic defects. Clearly, these changes in the surface composition and surface chemistry are a hindrance to the analysis of real materials. A review of the basic phenomena and their manifestation during AES analysis is presented.

Owing to the widespread concern for sample damage during AES analysis, a task group has been established within the Auger subcommittee of ASTM. This group is charged with the compilation and distribution of practical information about electron beam damage during AES. The results of a survey carried out by this group will also be presented highlighting, in particular, (1) real materials prone to electron beam damage (2) the instrumental parameters associated with the damage and (3) recommended practices, if any, for alleviation of the problem.

Thursday, 8:30 am

C-1

The Second Symposium on Applied Surface Analysis

June 11-13, 1980

University of Dayton
Dayton, Ohio 45469

Title: "Application of Rutherford Backscattering to Analysis of Glasses"

Authors: F. Ohuchi and P. H. Holloway
Department of Materials Science and Engineering
University of Florida, Gainesville, FL 32611

Abstract:

Rutherford backscattering has been widely used to investigate material phenomena, such as impurity solution and diffusion, epitaxial layers and oxidation, etc. The technique is unique in that microanalytical depth profiles can be obtained without sample erosion or layer removal.

Both proton and helium backscattering has been used in this study to investigate thin and thick glass films. The advantages and disadvantages of each particle will be illustrated for glass analysis. The incorporation of sodium into thin glass films, ion exchange reactions, and potassium migration during electron irradiation have all been studied with the backscattering technique. The data show that very little sodium is retained in rapidly quenched glass; slow cooling aids the retention of sodium in the glass. Upon exposing soda-silicate glass to molten KNO_3 , an ion exchange reaction converts the glass surface to a potassium-rich phase. Upon subsequent exposure to 2 keV electrons, the potassium is observed to diffuse into the glass bulk, due to field enhanced diffusion. The backscattering data will be compared to Auger electron data to illustrate the problems associated with analysis of glass surfaces.

Thursday, 9:20 am

C-2

PRINCIPAL COMPONENT ANALYSIS OF AUGER LINE SHAPES AT SOLID-SOLID INTERFACES

By

Stephen W. Gaarenstroom
Analytical Chemistry Department
General Motors Research Laboratories
Warren, Michigan 48090

ABSTRACT

The analysis of Auger line shape changes during a compositional depth profile can be used to determine the existence of a distinct chemical state at a solid interface. The procedure involves principal component analysis of a data matrix made up of selected Auger spectra from the depth profile. The number of chemical components in the overlayer, interface region, and underlayer is equal to the number of non-zero eigenvalues in the data matrix. Because spectra contain noise, some criterion must be used to decide when an eigenvalue is statistically different from zero. Provided reference spectra of all chemical components are available, the eigenvector solution can be rotated to give quantitative information on each component. The procedures described in this work are more rigorous than the visual inspection of Auger line shapes and are less time consuming than spectral stripping or curve resolving procedures.

Line shape analysis was applied to Cu MVV and Cu LMM Auger spectra from cuprous oxide heterojunction photocells. A layer of Cu metal was found to be present at the interface of a SnO_2 -doped CdO heterojunction photocell.

Thursday, 9:40 am
C-3

**AES Depth Profiling of Trace Elements in a Multiple
Transition Metal System Using Spectrum Subtraction
Techniques.**

G. E. Hammer

Goodyear Tire & Rubber Co
Fiber Reinforcement Technology
1144 East Market Street
Akron, Ohio 44316

Auger peak overlap among the transition metals can seriously interfere with obtaining accurate depth profiles from multi-element systems, particularly of low concentration constituents. There are many cases in the analysis of alloys, corrosion products, and metallurgical coatings where in-depth profiles of trace elements may be of interest. Therefore, the development of a technique to extract such profiles from the available data would be desirable.

In this paper, we present a method which was developed to obtain AES depth profiles of trace amounts of Group VIII elements in a brass matrix but which would be applicable to other systems. Spectrum subtraction techniques are applied to raw profile data to obtain a trace element profile corrected for peak overlap with the matrix Auger lines. The correctness of assumptions implicit in the technique is discussed, and the AES results obtained are compared with simultaneous SIMS profiles.

Thursday, 10:00 am

C-4

CHARACTERIZATION, GROWTH AND MECHANICAL PROPERTIES OF LAMINATE FILMS

Robert W. Springer
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ABSTRACT

The production of metal coatings of high strength and excellent surface finish by vapor deposition methods is dependent on many "new" parameters. The usual parameters varied during deposition are temperature, rate of deposit, angle of incidence, and in the case of sputtering, various biasing techniques. A study of gas interaction with growing films allowed the quantification of residual gas spectra observed in the chamber in terms of the impurity content. Auger Electron Spectroscopy was used to determine various elemental concentrations over two orders of magnitude. As a result, when the sticking coefficient of a gas on a clean surface is known as a function of temperature and coverage, the impurity content within the film may be computed.

Using this result, a pulsing technique known as the Pulsed Gas Process (PGP) was developed to put known amounts of impurity into the growing deposit. The use of oxygen in aluminum produces high strength specular films into the 25 μ range and thicker. The strength is adjustable from 3 kpsi to 60 kpsi by varying the pulse spacing. The use of carbon in tantalum films also produces high strength. However, the auger depth profiling did not show the same results as observed for oxygen in aluminum. When the PGP was applied to a curved surface, a new set of parameters was found that allow shapes to be produced.

In order to understand the nature of the films and the growth process, Auger Electron Spectroscopy, ESCA, Transmission Electron Microscopy, Scanning Electron Microscopy and X-Ray diffraction were used to obtain a composite picture of the growth process. The results of this analysis for aluminum and tantalum films will be presented and discussed.

Thursday, 10:40 am

C-5

Protective and Non-Protective Oxide Formation On 304 Stainless Steel

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Parameters controlling the formation of protective and non-protective oxides on 304 stainless steel were examined by using Auger electron spectroscopy to monitor oxides formed in the vacuum chamber. Variables found to influence the oxide formation include: oxygen partial pressure, the location of the oxide over a grain or grain boundary, sample grain size, and anneal treatments. Low pressure oxidation ($<10^{-5}$ Torr oxygen pressure) at 800°C produced a thin chromium oxide on annealed samples with 40µm grain size; this thin ($\sim 200\text{\AA}$) protective oxide did not grow with 15 minutes additional oxidation in air at 800°C. In contrast, initial oxidation of the same annealed material at oxygen partial pressures greater than 10^{-2} torr caused the formation of thicker iron-rich oxides. The oxides formed at these higher pressures were often non-uniform having thin chromium-rich oxides near grain boundaries and thicker iron-rich oxides over the grains. The oxide formed over smaller grained material ($\sim 4\mu\text{m}$ grain size) was chromium rich for all conditions examined. Annealing treatments influenced oxidation by varying the amount of chromium available at the surface for oxide formation. At temperatures above 800°C chromium evaporates from the sample decreasing the amount of chromium on the surface and altering the chromium profile into the metal. If carbon is present on the sample surface near 500°C a chemically induced chromium segregation occurs (similar to grain boundary sensitization) which also severely alters the chromium distribution in the near surface region of the material.

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Thursday, 11:30 am

C-6

Surface Segregation in Hot-Dip Metallic
Coatings on Steel Substrates

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ABSTRACT

Using Auger spectroscopy, minor alloy additions to the coating metal have been found to be enriched at the surface of zinc and aluminum coated steels. This enrichment could explain the improved corrosion resistance previously found for these alloy coatings. After the original surface had been removed, the reseggregation of the minor constituents was investigated as a function of temperature for air and vacuum environments. Possible consequences of this diffusion to the surface of these materials will be discussed for typical applications.

Thursday, 11:50 am

C-7

"ESCA Studies of Polymer Surfaces"

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The range of properties which can now be built into a polymer system is enormous and this coupled with the techniques which are available for the chemical, physical, electrical and mechanical modification of surface and bulk properties provides almost unlimited scope for tailor making materials for particular applications. The emphasis therefore over the past 15 years or so in polymer science has shifted from the chemistry of polymerization processes to the processing of polymers. Since many of the important features in this respect are determined by the structure, bonding and reactivity of the outermost few tens of angstroms, the motivation for applying the methods of surface science to polymers is not difficult to understand. In addition to the self evident technologically (and industrially) important areas of the surface science of polymers such as those involved in lamination, printing, weathering and segregation of additives the development of organic based thin film device technology in the past few years has been materially aided by the detailed insights provided by the shots in the surface scientists locker. ESCA provides the single most powerful tool for elaborating details of structure and bonding in the surface regions of polymers.

The wide range of available information levels endows the technique with wide ranging capabilities and examples will be described of ESCA studies of migration phenomena, the synthesis of thin polymer films by plasma techniques, (of interest in a wide variety of fields including microelectronics), the nitration of cellulose (of interest in the propellants and explosives fields), the oxidative functionalisation of surfaces (of interest in lamination, weathering and printing fields).

Friday, 8:30 am

E-1

ON THE THICKNESS AND SPATIAL DISTRIBUTION
OF A FLUOROPOLYMER FILM DEPOSITED BY SOLUTION DIPPING

by

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ABSTRACT

1H-1H-pentadecafluorooctyl methacrylate is essentially a methyl methacrylate polymer with a fluorocarbon side chain substituted for one of the methyl hydrogens. It is applied by dipping the part into a solution, with Freon TF as the solvent and the fluorocarbon polymer as the solute.

The purpose of this work is to consider the spatial distribution of the resulting film of barrier compound when it is deposited in this manner. Auger electron spectroscopy, X-ray photoelectron spectroscopy and a quartz crystal monitor were used and the specific variables considered are concentration and withdrawal velocity.

There exists a critical withdrawal velocity (dependent on concentration) above which the film is nonuniform and below which the film is macroscopically uniform. Below the critical velocity, the thickness varies with velocity with approximately a $v^{2/3}$ dependence. For macroscopically uniform films, a microscopic nonuniformity exists with a coverage of about 1/4 for an average film thickness of 90 Å.

Friday, 9:20 am

E-2

The use of Electronic devices in an attempt to stimulate
catalytic reactions.⁺

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A new attempt has been made to study the behaviour of catalytic reaction under electron excitation influences^(1,2). Therefore, simple reaction as CO oxidation has been mainly investigated using hot electron beam from large surface MIS tunneling diodes with platinum films as top collector (Si-SiO₂-Me). A parallel study of the same reaction using electron emission into vacuum from platinum thin films with discontinuous structure⁽³⁾ was also achieved in order to compare both ways of excitation. The design of the platinum island structure devices is reported. Results recorded with this two types of devices show the CO oxidation reaction rate versus the applied pulsed bias voltage. Their discussion is made in terms of the enhancement of catalytic activity, electron transfert mechanism and the role played by tunnel electrons (possible thermal effect is taken into account).

(1,2). W. Haidinger and J. Figar, Chem. Phys. Lett., Vol. 11, n°5
545 (1971) and Vol. 19, n° 4 564 (1973).

(3) G. Dittmer, Thin Solid Film, 9 317 (1972).

⁺Work supported by the IRIS (Institute for Research in Interface Science) Program sponsored by the Belgian Ministry of Science Policy.

⁺⁺Postdoctoral IRSIA Fellowship. Working also in Laboratoire de Chimie Inorganique, Université de l'Etat à Mons, 23, avenue Maistriau, 7000 Mons - Belgium.

Friday, 9:40 am

E-3

"THE KINETICS OF THE INITIAL OXIDATION
OF NICKEL USING RESISTIVITY MEASUREMENTS"

By:

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ABSTRACT

The rate determining step during the initial oxidation of nickel can be determined using resistivity measurements. Two thin nickel foils are placed in separate quartz tubes in the hot zone of a furnace. Both are annealed in H_2 to reduce the existing oxide. Then one sample is exposed to a set compared to that of the sample remaining in H_2 . These resistance changes are measured by applying a constant current and measuring the voltage change as the oxidation proceeds. Experiments were performed over a temperature range of 600-900°C and at a set oxygen partial pressure ranging from 0.01-0.5 atmospheres. The rate determining step can then be determined as a function of Temperature and Oxygen Partial Pressure.

Friday, 10:00 am

E-4

INVESTIGATION OF BONDING AND ADHESION PHENOMENA
BY COMBINED ISS/SIMS ANALYSIS

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and
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ISS and SIMS are extremely useful complementary techniques for the investigation of bonding and adhesion phenomena. Unbonded adhesive and substrate surfaces are characterized with relative ease for overall composition, preparation-induced compositional changes, handling and processing contaminants, and elemental components which serve as natural identification "tags." Bond surfaces from failed bonded assemblies present a more difficult task. Failures resulting from a multitude of factors can occur at many points within the adhesive/adherend interphase. Multi-layer adhesive systems introduce additional complexities. Most often, the need for chemical information is vital to a detailed surface investigation in bonding applications.

Analysis results are presented from investigations on materials for bonding and failed bond surfaces encompassing controlled laboratory studies, production line applications, and actual field failures. Specific examples from both metal-to-metal and rubber-to-metal bonded components illustrate failures occurring at loci in the adhesive layer, in the metal substrate oxide layer, and at the substrate/adhesive interface. Other combined ISS and SIMS data are discussed which verify adhesive application, pinpoint bond separation time, and help elucidate bond failure nature, mechanisms, and bond surface chemistry.

Friday, 10:40 am

E-5

"Structure and Properties of Organosilane Primers for Adhesive Bonding" by F. J. Boerio and J. W. Williams, Department of Materials Science and Metallurgical Engineering, University of Cincinnati, Cincinnati, Ohio 45221.

External reflection infrared spectroscopy was used to determine the structure of films formed by the adsorption of γ -aminopropyltriethoxysilane (γ -APS) onto iron mirrors from aqueous solutions. Films formed at the natural pH of 1% aqueous solutions (10.4) were composed of oligomers characterized by infrared bands near 1550, 1480, and 1110 cm^{-1} while films formed at pH values less than about 9.0 were composed of polysiloxanes characterized by bands near 1600, 1500, and 1135 cm^{-1} . The polysiloxanes formed at pH values less than 9.0 were much more effective as primers for improving the hydrothermal stability of iron-epoxy lap joints than the oligomers obtained at pH = 10.4. Joints prepared from substrates primed with γ -APS at pH = 8.0 retained about 80% of their strength after immersion for 60 days in water at 60°C while those prepared from substrates primed at pH = 10.4 retained only about 45% of their strength. By comparison, joints prepared from unprimed substrates retained only about 30% of their strength after such exposure. This is the first time that the molecular structure of an adsorbed silane has been related to its performance as a primer for adhesive joints.

Friday, 11:00 am

E-6

THIN ANODIC OXIDE FILMS ON ALUMINUM ALLOYS
AND THEIR POTENTIAL CORROSION CHARACTERISTICS

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and

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The full utilization of aluminum alloys in metal-to-metal adhesively bonded structures requires a thorough knowledge of all the factors that go into the fabrication of a bonded joint. One of the major factors that must be considered in the study of adhesive bonding technology is the surface preparation of the metal adherend.

Utilizing surface analysis techniques, the complete chemical signature of a metal surface can be obtained on the (1) as-received material; (2) chemically cleaned surface; (3) anodic oxide layers and, (4) protective qualities of the anodic oxide layer with respect to corrosion.

This type of information is required in order to build a data base from the surface of each metal alloy. Reasonably sound judgements can then be made as to the influence of these surfaces on the long-term properties of a bonded structure.

Friday, 11:20 am

E-7

"PHOSPHATE-FLUORIDE ETCHING EFFECTS ON TITANIUM ALLOY SURFACES"

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The effects on the surfaces of seven titanium alloys treated with an industrial phosphate-fluoride "conversion" treatment were characterized by scanning electron microscopy (SEM), Auger electron spectroscopy (AES), X-ray photo-electron spectroscopy (XPS) and secondary ion mass spectroscopy (SIMS). Generally this treatment produces a thick oxide layer containing fluorine throughout. The Ti_{LMV} Auger spectra from these oxide layers differed slightly in shape from pure TiO_2 . Positive ion SIMS detected fluorine species such as TiF^+ , TiF_2^+ , and $TiOF^+$.

The chemical state of Ti, O, F and Na was investigated with XPS. Since there was uncertainty as to charging and the chemical state of C, the C_{1s} was not used as a calibration for determining binding energy shifts. A more reliable method of measuring real binding energy shifts was to rely on binding energy differences between Ti and O because charging effects would be the same for both peaks and changes in the absolute binding energy difference, compared to TiO_2 , reflect real chemical differences between the fluorine containing surface oxide and pure TiO_2 .

The XPS data indicates that surface fluorine is chemically bound to surface contaminants, ie., Na, O and C. Within the bulk oxide, fluoride is chemically bound to both oxygen and titanium forming an oxy-fluoride. Data is presented which correlates this oxy-fluoride layer with the locus of failure in fluoride treated adherend adhesive bonded structures.

Friday, 11:40 am

E-8

SECTION V

SYMPOSIUM CONCLUSIONS

The purpose for holding this Symposium was to bring together basic research, applied research and problem solving efforts all involving some type of surface analysis of Air Force interest into one meeting to try to improve communications and understanding of persons in these various endeavors. That a need for such a meeting exists seems, more and more, to be established from the many favorable comments received and from the growing interest in this Symposium.

The Air Force Office of Scientific Research is to be commended for supporting this Symposium.

